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IS 12008 (1986): Silica Gel, Chromatographic Grade [CHD 1: Inorganic Chemicals]



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Indian Standard

SPECIFICATION FOR SILICA GEL,
CHROMATOGRAPHIC GRADE

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SILICA GEL, CHROMATOGRAPHIC GRADE

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Indian Standard

SPECIFICATION FOR SILICA GEL, CHROMATOGRAPHIC GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 November 1986, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 Silica gel is prepared generally by gelling sodium silicate solution with either hydrochloric acid or sulphuric acid. However in recent practice, it is prepared by passing solution of sodium silicate through cation resin bed and gellifying pure silicic acid solution by suitable gellifying agents. Gellification is achieved by close control of operating parameters, such as pH, concentration, temperature, etc, in order to obtain silica gel of consistent quality.

0.3 Chromatographic grade silica gel is mainly used for analytical purposes in thin layer chromatography, high performance liquid chromatography and partition column chromatography. Silica gel used as industrial desiccant is covered in IS : 3401-1979*.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard presents the requirements and the methods of sampling and test for chromatographic grade silica gel.

*Specifications for silica gel (*second revision*).

†Rules for rounding off numerical values (*revised*).

2. GRADES

2.1 Material shall be of the following two grades:

- a) *Grade 1* — For thin layer chromatography,
- b) *Grade 2* — For partition column chromatography, mainly 250/125 micron IS sieve or 150/75 micron IS sieve.

3. TYPES

3.1 Grade 1 of the material shall be of two types:

- a) *Type 1* — shall be silica gel without binder,
- b) *Type 2* — shall be silica gel with binder.

4. REQUIREMENTS

4.1 The material shall consist of silica gel in granular form of various sizes for partition column chromatography and for thin layer chromatography.

4.2 The material shall also comply with the requirements given in Table 1 for thin layer chromatography and Table 2 for partition column chromatography, which tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of Table 1 and col 4 of Table 2.

5. PACKING AND MARKING

5.1 **Packing** — The material shall be packed in clean, dry and air-tight bottles not exceeding 250 g. The container shall not be opened until required for use.

5.2 Each container shall bear legibly and indelibly the following information:

- a) Name, grade, type and size of the material;
- b) Name of the manufacturer and/or his recognized trade-mark, if any;
- c) Mass of the material in the container;
- d) Date of packing; and
- e) Batch number.

TABLE 1 REQUIREMENTS FOR SILICA GEL FOR THIN LAYER CHROMATOGRAPHY

(Clause 4.2)

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF To CL No. IN APPENDIX A)
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Particle size passing through 50 micron IS sieve, percent by <i>Max</i>	97	97*	A-2
ii)	pH of aqueous suspension (10 percent)	5.5 to 7.0	6.0 to 7.5	A-3
iii)	Sodium (as Na), ppm, <i>Max</i>	700	—	A-4
iv)	Chloride (as Cl), ppm, <i>Max</i>	200	200	A-5
v)	Iron (as Fe), ppm, <i>Max</i>	100	100	A-6
vi)	Loss on ignition, percent by mass, <i>Max</i>	20	—	A-7
vii)	Gypsum, percent by mass	—	12 to 14	A-8
viii)	Suitability test	shall pass the test	shall pass the test	A-9

*Wet sieve test.

TABLE 2 REQUIREMENTS FOR SILICA GEL FOR PARTITION COLUMN CHROMATOGRAPHY

(Clause 4.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF To CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Particle size distribution between any mesh (250/125 micron or 150/75 micron IS sieve), <i>Min</i>	95	A-2
ii)	pH of aqueous suspension	5.5-7.0	A-3
iii)	Sodium (as Na), ppm, <i>Max</i>	700	A-4
iv)	Chloride (as Cl), ppm, <i>Max</i>	200	A-5
v)	Iron (as Fe), ppm, <i>Max</i>	100	A-6
vi)	Suitability test	To pass the test	A-9

5.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6. SAMPLING

6.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

A P P E N D I X A

(*Clause 4.2; and Tables 1 and 2*)

METHODS OF TEST FOR SILICA GEL, CHROMATOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PARTICLE SIZE

A-2.1 For Thin Layer Chromatography

A-2.1.1 Procedure — Weigh accurately 50 g of the sample and place it over 50 micron IS sieve. Shake it for 15 minutes. The mass of material passing through the sieve gives the particle size of the sample expressed as percentage by mass.

A-2.1.2 Calculation

$$\begin{array}{l} \text{Material passing through 50 micron} \\ \text{IS sieve, percent by mass} \end{array} = \frac{M_1 - M_2}{M_1} \times 100$$

*Specification for water for general laboratory use (*second revision*).

where

M_1 = mass of material taken for sieving, and

M_2 = mass of material retained over the sieve.

A-2.2 For Partition Column Chromatography

A-2.2.1 Apparatus

A-2.2.1.1 Sieve — 125 micron and 250 micron IS sieve (or 75 micron and 150 micron IS sieve.).

A-2.2.2 Procedure — Take accurately about 100.00 g of material of 250 micron/125 micron IS sieve size and sieved through 250 micron sieve and material passing through 250 micron sieve is sieved through 125 micron sieve, quantity retained on 125 micron sieve must not be less than 95 g. In other words, mass of material retained on 250 micron sieve plus mass of material passing through 125 micron IS sieve should not be more than 5 g.

A-3. pH VALUE

A-3.1 Apparatus

A-3.1.1 pH Meter

A-3.2 Procedure — Prepare an aqueous suspension of the material by shaking for 10 minutes, 5 g of the material with 50 ml of water (free from ammonia and carbon dioxide) in a clean polyethylene flask. Decant the supernatant liquid and measure pH by using any standard pH meter which is standardized against standard buffer solution of pH 4.0 and pH 9.2.

A-4. SODIUM AND ITS COMPOUNDS

A-4.1 Apparatus

A-4.1.1 Flame Photometer

A-4.2 Reagents

A-4.2.1 Concentrated Nitric Acid — See IS : 264-1976*.

A-4.3 Procedure — Weigh 0.5 g of the sample into a 250 ml glass dish previously washed with dilute nitric acid. Add 10 ml of water and 10 ml of concentrated nitric acid and evaporate the contents to dryness on a hot plate. Add another 10 ml of concentrated nitric acid and evaporate to dryness again. Extract the residue with 20 ml of nitric acid (1 : 1) by gentle boiling. Filter through Whatman No. 40 or equivalent filter paper into a 250-ml volumetric flask. Wash three times with hot water. Cool

*Specification for nitric acid (second revision).

the flask and dilute to the mark with water. Take 25 ml of the solution and dilute to 100 ml in a volumetric flask. Flame the diluted solution using a flame photometer. The instrument shall be calibrated in the range of 0 to 5 mg/l of sodium.

A-4.4 Calculation

Sodium and its compounds (as Na_2O), percent by mass = $X \times 0.2$

where

X = mg/l of Na_2O from the calibration curve.

A-5. WATER SOLUBLE CHLORIDES

A-5.1 Reagents

A-5.1.1 Standard Chloride Solution — Prepared by adding 28.2 ml of N/10 hydrochloric acid to water to produce 100 ml.

A-5.2 Procedure

Boil 0.5 g (accurately weighed) with a mixture of 50 ml water and 1 ml of dilute nitric acid (approximately 5 N), Cool, filter and to the filtrate add 1 ml of silver nitrate solution (0.4 percent).

Any opalescence produced shall not be greater than the standard opalescence as stated in **A-5.2.1**.

A-5.2.1 One millilitre of the solution is equivalent to 0.1 mg of chloride (as cl). To 50 ml of water add 1 ml of standard chloride solution, 1 ml of dilute nitric acid and 1 ml of silver nitrate solution. Mix after each addition, and use as a comparison standard after 5 minutes.

A-6. IRON

A-6.1 Reagents

A-6.1.1 Dilute Hydrochloric Acid — 1 : 1 (v/v).

A-6.1.2 Standard Potassium Permanganate — 0.1 N.

A-6.1.3 Ammonium Thiocyanate Solution — 57 percent.

A-6.1.4 Standard Iron Solution — Dissolve 8.65 g of ammonium ferric sulphate in 50 ml of concentrated nitric acid and dilute to 1 000 ml. One ml of this solution is equivalent to 0.01 mg of iron (as Fe).

A-6.2 Procedure

Boil 0.1 g with a mixture of 10 ml water and 1 ml of dilute hydrochloric acid, cool and filter.

Take filtrate and add 1 drop of N/10 potassium permanganate and mix. Add 5 ml of ammonium thiocyanate solution (570 g/l) and 10 ml of mixture of equal volumes of amyl alcohol and amyl acetate: shake vigorously and allow to separate.

Take 1 ml of standard solution of iron, add 1 ml of dilute hydrochloric acid and dilute with water to the same volume as acidified solution of the test sample. Add 1 drop of N/10 potassium permanganate and from this point follow the procedure described for sample and compare colour with standard. The colour produced by the sample is less than colour produced by standard if iron content in sample is less than 100 ppm.

A-7. LOSS ON IGNITION

A-7.1 Apparatus

A-7.1.1 Platinum Crucible

A-7.2 Procedure

Take 1 g of the material in a clean, dry platinum crucible. Keep the crucible and its contents on a hot plate for 10 minutes. Heat further at 550°C for 5 minutes and finally at 1100°C for 30 minutes. Cool, weigh and calculate the percent loss on ignition.

A-7.3 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = Loss in mass in g, and

M_2 = Mass in g of the sample taken for the test.

A-8. GYPSUM

A-8.1 Reagents

A-8.1.1 *Dilute Ammonia Solution* — 25 percent.

A-8.1.2 *Standard EDTA Solution* — 0.1 M.

A-8.1.3 *Eriochrome Black T Indicator*

A-8.2 Procedure — Take about 1 g of the material in a 250-ml beaker and mix with about 150 ml of water and stir well with a glass rod. Filter through a double layer filter paper No. 41 after 30 minutes. Wash 5 times with water. Add 2 ml of dilute ammonia solution in the filtrate and titrate against standard EDTA solution using eriochrome black T as indicator.

A-8.3 Calculation

Gypsum (as $\text{CaSO}_4 \cdot 5\text{H}_2\text{O}$), percent by mass = $\frac{A \times 1.45}{M}$

where

A = volume in ml of standard EDTA, and

M = mass in g of sample taken for the test.

A-9. SUITABILITY TEST

A-9.1 Suitability for Thin Layer Chromatography

A-9.1.1 Reagents

A-9.1.1.1 Solvent mixture — See A-9.2.1.1.

A-9.1.1.2 Toluene

A-9.1.2 Procedure — Make 30 g into slurry with about 60 ml water. Spread five 20×20 cm plates and allow the slurry to dry in air. The material should be smooth and free from hollows. Developed plates using toluene after spotting with test mixture should give clear separation with colour in straight lines horizontally and vertically. Spray plates with 2N H_2SO_4 in alcohol and heat at 110°C for 30 minutes. Very few black particles shall be observed.

NOTE — Time taken for solvent to travel 20 cm of length of plate shall be about 30 minutes.

A-9.2 Suitability Test For Partition Column Chromatography

A-9.2.1 Reagents

A-9.2.1.1 Solvent mixture — Mix 10 mg of 4 methoxy-azo benzene with 10 mg each of Sudan yellow and Sudan red. Dissolve this, mix in 20 ml of the solvent prepared by mixing 4 parts of petroleum ether with 1 part of benzene.

A-9.2.2 Procedure — Prepare a column of 5 cm length using petroleum ether. Use column of 1.5 cm diameter. Add 5 ml of solvent mixture, wash the column with 10 ml of the same solvent. The order of the absorption of three different dyes shall be as under:

In Column

Yellow

Yellow

Order of Elution

- 1) 4-Methoxy-azo benzene—
solution is yellow
- 2) Sudan yellow—solution is
orange yellow
- 3) Sudan red—solution is red
in colour.

APPENDIX B

(Clause 6.1)

SAMPLING OF SILICA GEL, CHROMATOGRAPHIC GRADE**B-1. SCALE OF SAMPLING**

B-1.1 Lot — All the containers in a single consignment of silica gel of the same type and for the same use (for thin layer chromatography or for partition column chromatography) drawn from a single batch of manufacture shall constitute a lot.

B-1.2 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

B-1.3 Unless otherwise agreed to between the buyer and the seller, the number of containers to be chosen from the lot shall depend upon the size of the lot and shall be as given in Table 3.

TABLE 3 SCALE OF SAMPLING

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
Up to 25	3
26 to 50	4
51 to 100	5
101 to 150	7
151 and above	10

B-1.4 The containers shall be selected from the lot at random and in order to ensure the randomness of selection, the method given in IS : 4905-1968*.

B-2. TEST SAMPLE AND REFEREE SAMPLE

B-2.1 From each of the containers selected draw approximately 150 g of silica gel with help of suitable sampling implements, the material drawn from different containers shall be mixed thoroughly to give a composite sample weighing about 500 g.

B-2.2 The composite sample shall then be divided into three parts, one for the purchaser, another for the supplier and the third for the referee. These parts shall be transferred to separate containers which shall be suitably closed and marked with all the details of sampling.

*Methods for random sampling.

B-2.3 The referee sample shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute.

B-3. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-3.1 All the sample containers shall be visually examined for the requirements given in 4.

B-3.2 Tests for all the characteristics given in Table 1 or Table 2 shall be conducted on composite sample.

B-3.3 The lot shall be declared as conforming to the requirements of the specification if all the containers pass the requirements of 4 and the composite sample satisfies the relevant requirements given in Table 1 or Table 2.